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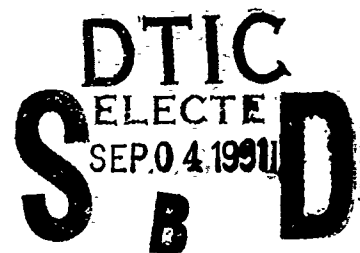
RL-TR-91-118

Final Technical Report
July 1991

CHARACTERIZATION OF ELECTRONIC MATERIALS

MANLABS, Inc.

Edward P. Warekois



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13. ABSTRACT (Maximum 200 words) This report describes development and implementation of experimental techniques to fabricate specimens from new or novel materials developed under several Air Force programs. X-ray orientation, optical polishing and metallurgical processing are discussed. Specimen materials include hydrothermally grown quartz crystals, non-oxide based glasses, indium phosphide crystals, bismuth containing electro-optic materials and several other miscellaneous materials.					
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SUMMARY

This report covers materials, services and investigations provided under contract F19628-87-C-0125 over the time period from 01 May 1987 to 30 April 1990.

The principal effort under this contract was directed to the development and implementation of experimental techniques to fabricate specimens from new or unique electronic materials developed under several Air Force programs. Such specimens were optimized to obtain the parameters required to characterize the behavior and properties of these materials.

The contract was concerned with the hydrothermal growth of single crystal of quartz ;with several classes of non-oxide based glasses being developed for IR applications ;with the growth and processing of indium phosphide crystals for use as semi-conductor devices and with the growth and characterization of bismuth containing electro-optic materials for optical components in advanced optical signal processing systems.

The requests for services were submitted by the staff of the Solid State Material Directorate within RADC. The data ,the techniques and any conclusions relative the specimens were submitted as individual letter reports to the staff requestor. A total of 118 such letter reports were compiled. The type of service or fabrication requested with suitable sample identifications are tabulated in TABLE I.



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CHARACTERIZATION of ELECTRONIC MATERIALS

I. Introduction

The present contract has been in support of the Air Force RADC/ESMC materials laboratories effort in the programs concerned with the development of new or unique materials for the future application in the armed forces electronic based systems.

The services and tasks that were performed as part of this effort covered a multiple of technical disciplines and involved many experimental methodologies to characterize the parameters of the emerging materials. Many of the methods have been detailed in previous reports on file as technical reports (TR-) that summarize similar programs conducted for the Solid State Directorate (ESMO) within the Rome Air Development Center (RADC) of the Air Force System Command. Notable examples of such reports are:

- a) Physical Characterization of Electronic Materials.
TR-81-167 dated July 1981--- F19628-78-C-0060
- b) Characterization of Solid State Materials.
TR-84-136 dated June 1984--- F19628-81-C-0046
- c) Preparation of Electronic Materials.
TR-87-85 dated July 1987 ---F19628-84-C-0060

The present program concentrated on several investigations related to the development of unique classes of materials with electrical, optical and magnetic properties suitable or required in the design and operation of various electronic systems for the Air Force. The significant programs involved:

1) the development of hydrothermally grown quartz crystals that would have exceptionally stable characteristics when evaluated as frequency sources or timing devices;

2) the development of IR transmitting materials based on a non-oxide glass matrix in order to eliminate or reduce absorption bands in the infrared frequency ranges;

3) the development of the methodology for the growth of bulk semi-conductor crystals or the deposition of thin films of such semi-conductors to fabricate epitaxial alloy layers of these classes of materials;

4) the development of growth systems to produce single crystal of mixed oxides having properties that would permit the material to be used in optical frequencies signal processing or control systems.

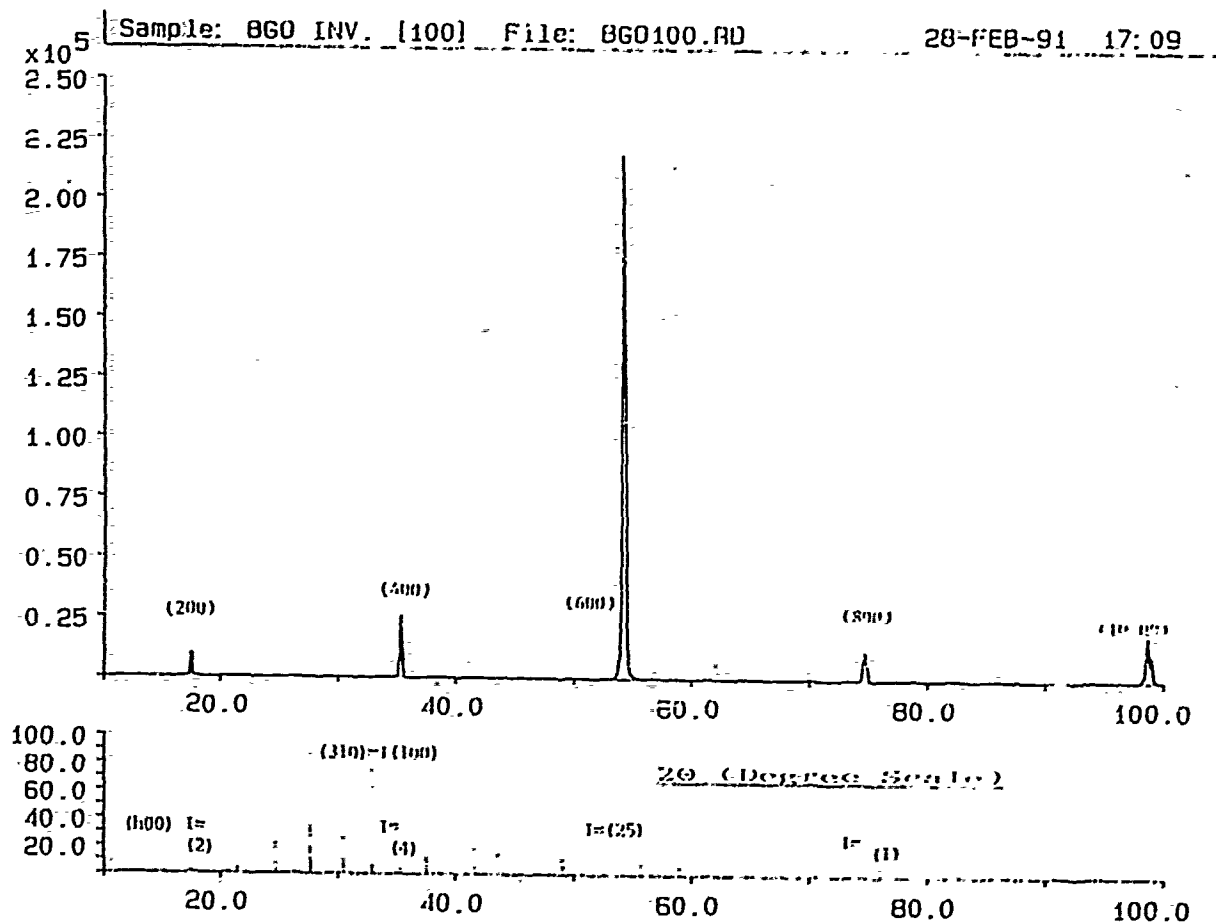
II. RESULTS and CONCLUSIONS

A list of the requested services submitted by the RADC staff is presented in TABLE I. The list includes the 118 individual projects that were completed and then reported as a letter report to the staff. Specific data or methods used are contained in the appropriate report. However, methods and technology utilized during this contract can be generalized and described as an experimental measurement that provides raw data as a recording, photograph or instrumental hardcopy print. Examples of such data used in the preparation of specimens under this contract are enclosed.

a) X-ray Diffraction Methods; The end product of the crystal growth programs were boules of the material with some form of growth feature on the surface of the boule, a facet or a cleavage plane. Under some conditions those topographic features are eliminated and one must use X-ray diffraction methods to obtain the crystallographic directions within the grown boule. The data can be either back-reflection Laue film records or diffractometer 2θ recordings. Features in the Laue film or angular positions of the diffraction peaks serve as the guide lines in the fabrication of known or oriented geometries.

A significant effort involved the fabrication of specifically oriented cubes from the bismuth/silicon/germanium oxide crystals, i.e. $\text{Bi}_{12}\text{SiO}_{20}$ (BSO) for use as active elements in holographic systems designed to scan optical characters. The cubes used for these elements required that orthogonal sets of crystallographic planes be prepared with one set of planes being the (100) family and the other sets being (110) sets of planes.

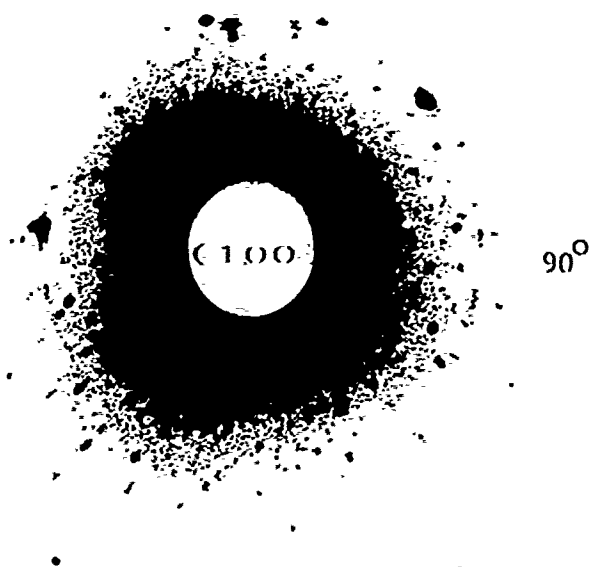
In order to produce such geometries from the grown boules without a significant loss of material, standard sets of Laue photographs were produced and the crystallographic features confirmed by diffraction scans that confirmed the d-value of the surface of interest. Once a Laue film has been referenced by using a combination of films and 2θ peaks, it is possible to then define any crystallographic direction without recourse to growth features or artifacts on the boule surfaces. Standard Laue films for the (100) and (110) BGO, along with the 2θ scans from those surfaces are shown as Figure (1) and (2).



0° 45°

FIGURE 1

- a) Diffractometer Scan of
BGO [100] Surface
 - b) Standard X-ray Pattern
Random Powder
 - c) Back-reflection Laue film
[100] Surface
- 110 Directions towards 0°
100 Directions towards 45°



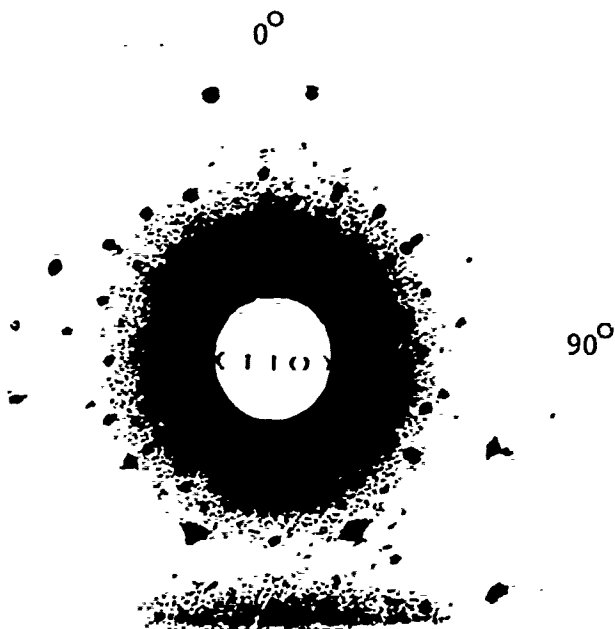
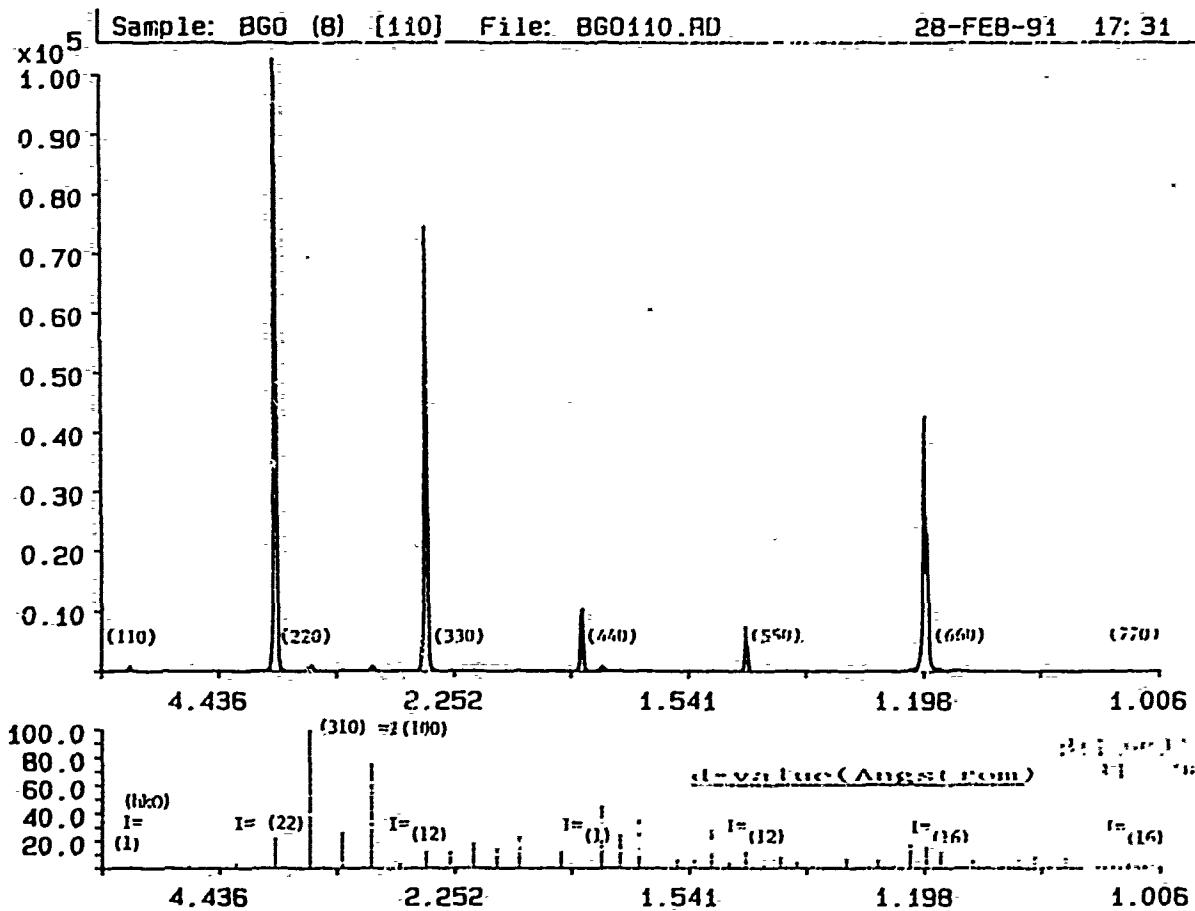


FIGURE 2

- a) Diffractometer Scan of BGO (110) Surface
 - b) Standard X-ray Pattern Random Powder (Note scale in d -values)
 - c) Back-reflection Laue film (110) Surface
- 110 Direction towards 0°
100 Direction towards 90°

b) Optical Fabrication; A significant portion of the requests listed in the table required some sort of cutting, grinding or polishing of specimens in order to define an optical or metallurgical surface. Most surfaces prepared for metallurgical inspection require only that those surfaces be flat, relatively smooth and clean of any artifacts that would mask the true features of the surface. The requirements and definition of an optical surface is much more critical. The smoothness, flatness and cleanliness are defined by standards. The details of such surfaces are examined using optical comparators, optical interferometers, surface topography profilers and comparison standards, commonly referred to as "Scratch/Dig" specifications.

Many of the applications under investigation at RADC involved information systems for the processing of optical signals. One such system involved the use of very smooth fused quartz plates arranged so as to act as a wave guide for an optical beam. Hence, the surfaces of such plates could not contain any feature or flaw that would introduce distortion into the optical beam. The stock quartz used for such plates was selected to have very few gross inclusions and a very uniform composition as defined by changes in the index of refraction. A further rigid requirement was that the optical surfaces produced from such plates would, also, have to be smooth and uniform over rather large areas. Several commercially available surface topographic microscopes have incorporated micro-processors into the optical systems so that such fine and exacting detail can be obtained from optical surfaces.

A typical surface obtained from a polished plate of Corning A-0 class material (a glass selected for no inclusion or non-uniformity) is shown as the output from such a surface topography system. Typical presentations from such surfaces are shown in Figure (3). These data which are both two and three dimensional plots of light scattered from a surface does give detail in the range required for the new optical systems. The scales on those print-outs do show difference in elevation and defines regions of inspection down to the nanometer ranges.

c) Machine Services: The requests that have been classified as "Mach.Serv." involved the use of machine shop facilities and staff. Typical requests included the design of small jigs and fixtures or several small assemblies to be used in larger experimental systems at RADC. In addition, to the more typical materials and methods found in most experimental machine shops, the services listed in the table have included specialized fabrications from boron nitride, high purity (OFHC) copper and the processing of extremely hard/brittle borides, carbides and many of the brittle semi-conductor crystals.

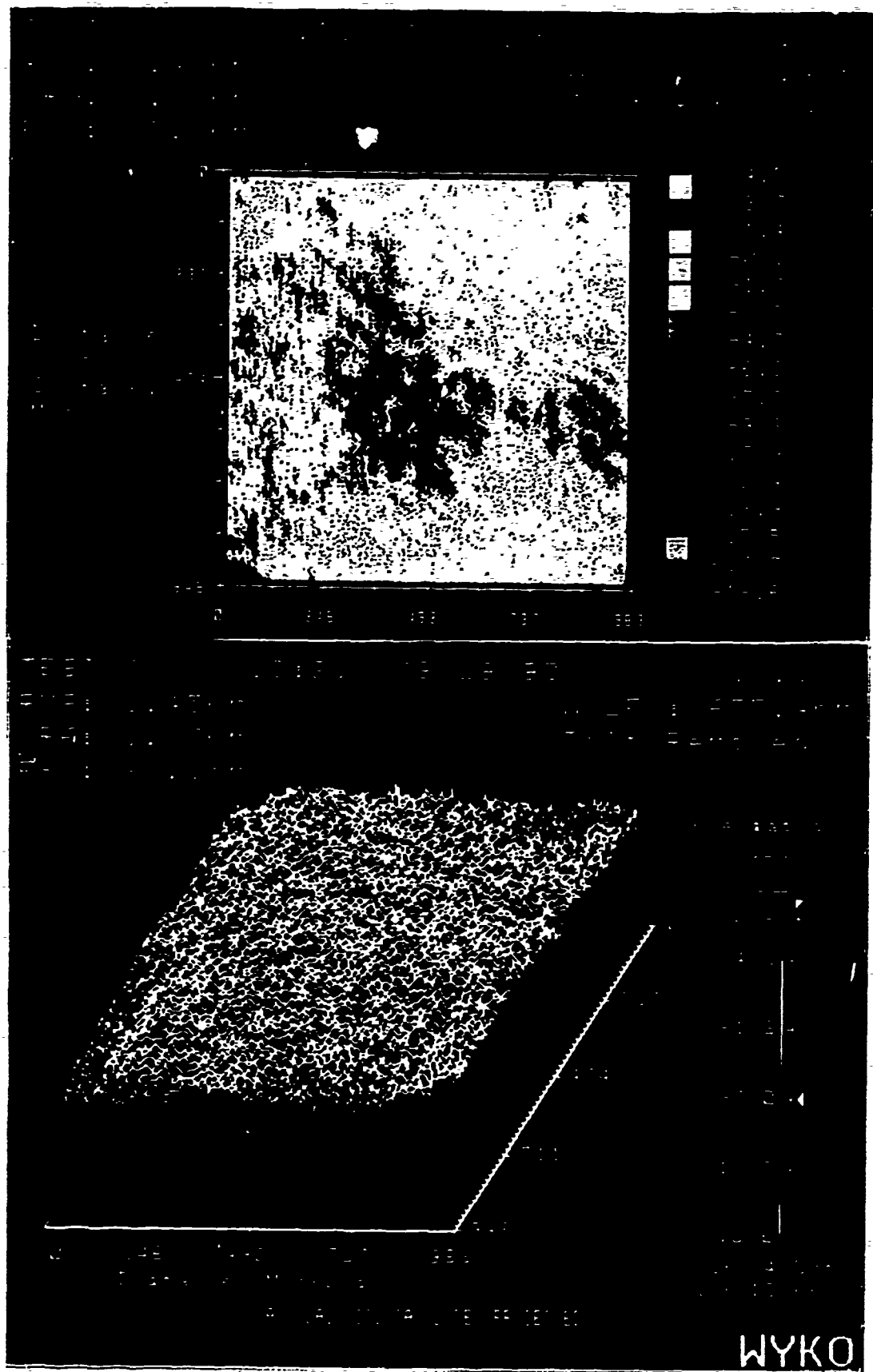


Figure 3 Two & Three Dimensional Surface Topography

TABLE 1List of Services and MaterialsContract F19628-87-C-012501 May 1987 to 30 April 1990

<u>ITEM NO.</u>	<u>RADC SR #</u>	<u>REQUESTOR</u>	<u>MATERIAL/SPECIMEN*</u>	<u>SERVICES**</u>
1.	5220	Golubovic	O: Cu Heat Sinks	Opt. Fab.
2.	5221	Harris	Q: QP-47	Opt. Fab.
3.	5222	Lipson	Q: QA 38M(b)	Opt. Fab.
4.	5223	Lipson	Q: E42-218A BHA #1	Opt. Fab.
5.	5224	Golubovic	O: Cu Heat Sink	Opt. Fab.
6.	5225	Lipson	Q: D14-45A #5	Opt. Fab.
7.	5226	Golubovic	O: Cu Heat Sinks (3)	Mach. Serv.
8.	5227	Lipson	Q: BHA-#1	X-ray Serv.
9.	5228	Euler	Q: X-67 M	Opt. Fab.
10.	5229	Lipson	Q: D14-45-36	Opt. Fab.
11.	5230	Wojtowicz	O: InP Xtal	X-ray Serv.
				Opt. Fab.
12.	5231	Golubovic	O: Copper Sink	Opt. Fab.
13.	5232	Drevinsky	O: Silicon	Back side polish
14.	5233	Golubovic	O: Cu Heat Sinks	Mach. Serv.
15.	5234	Lipson	Q: QA-SC-S & QA-SA-3	Opt. Fab.
16.	5235	Harris	Q: PRH252 & 262	Opt. Fab.
QPR #1				
17.	5236	Harris	Q: QS8	Opt. Fab.
18.	5237	Harris	Q: GC-26-39	Opt. Fab.
19.	5238	Harris	O: Graphfoil	Auger/SEM
20.	5239	Euler	Q: X81M	Mach. Serv.
21.	5240	Euler	Q: QA 50 Bar E	Opt. Fab.
22.	5241	Euler	Q: X67 Bar S of Stone M	Opt. Fab.
23.	5242	Harris	Q: X99 QS 11	Opt. Fab.
				ICAP/Spec.
24.	5243	Golubovic	O: Cu Heat Sink	Mach. Serv.
25.	5243A	Ahern	O: TiB ₂ Plate	Mach. Serv.
26.	5244	Harris	Q: FCZ & SWG B5	Opt. Fab.
27.	5245	Drehman	O: SrTiO ₃	X-ray Serv.
				Opt. Fab.
28.	5246	Euler	Q: X-81	Opt. Fab.
29.	5247	Golubovic	O: Cu Heat Sink	Mach. Serv.
30.	5248	Drehman	G: Rods	Opt. Fab.

QPR #2

31.	5249	Krambeck	G:	ZBLAN (13)	Opt. Fab.
32.	5250	O'Conner	Q:	2 sections	Mach. Serv.
33.	5251	Ahern	O:	TiB ₂ specimen	Mach. Serv.
34.	5252	Ahern	O:	TiB ₂ Xtals	High Pres. Services
35.	5253	Quinlan	O:	SrTiO ₃	Opt. Fab.
36.	5254	Lipson	Q:	Fe doped	Opt. Fab.
37.	5255	Harris	Q:	QS-13	Opt. Fab.
38.	5256	Hilton	O:	InP	X-ray Serv. Mach. Seeds
39.	5257	Harris	O:	InP	Mach. Serv.
40.	5258	Lange	O:	Cu heat sink	Opt. Fab.
41.	5259	Dumais	O:	MgO	Material & Opt. Fab.

QPR #3

42.	5260	Quinlan	O:	SrTiO ₃	Opt. Fab.
43.	5261	Quinlan	O:	SrTiO ₃	ICAP Emission
44.	5262	Quinlan	O:	SrTiO ₃	Opt. Fab.
45.	5263	Spaziani	O:	Fused (46)	Opt. Fab.
46.	5264	Dumais	O:	Plexiglass	Opt. Fab.
47.	5265	Quinlan	O:	SrTiO ₃	Opt. Fab. X-ray
Serv.					
48.	5266	Quinlan	O:	SrTiO ₃	Opt. Fab. X-ray Serv.
49.	5267	Dumais	O:	Film	Photography
50.	5268	Lange	O:	Cu heat sink	Material Mach. Serv.
51.	5269	Harris	O:	OA67X/OA67Z	Opt. Fab.
52.	5270	Murin	O:	Heat sink	Opt. Fab.
53.	5271	Lipson	O:	Fe doped H29-14N40E	Opt. Fab.
54.	5272	Quinlan	O:	SrTiO ₃	ICAP Emission

QPR #4

55.	5273	ElBayoumi	G:	WL#1 & SI#6	Opt. Fab.
56.	5274	Ahern	O:	TiB ₂	Mach. Serv.
57.	5275	Brown	G:	#130/81/82	Opt. Fab.
58.	5276	Brown	G:	#1361/2/3/4 67/74/78/79	Opt. Fab.
59.	5277	Lipson	O:	HgS-thin section	Opt. Fab.
60.	5278	Lipson	O:	HgS-thin section	Opt. Fab.
61.	5279	Brown	G:	#1339	Opt. Fab.
62.	5280	Ahern	O:	TiB ₂ & piston assembly	Mach. Serv.
63.	5281	Brown	G:	#1233	Opt. Fab.
64.	5282	Drehman	O:	High pressure processing	Press Serv.
65.	5283	Harris	O:	BSO Boules	X-ray Serv.
66.	5284	Bouthilette	O:	Glass	Opt. Fab.

QPR #5

67.	5285	Lipson	Q: BHA #2	Opt. Fab.
68.	5286	Harris	O: BSO	Chem. Analysis
69.	5287	Weeks	G: Salt Window	Opt. Fab.
70.	5288	Dumais	Q: Natural Synthetic Quartz	X-ray Serv.
71.	5289	Brown	G: #1023	Opt. Fab.
72.	5290	Lipson	Q: D14-45 40A #6	Opt. Fab.
73.	5291	Lipson	O: HgS-thin section	Opt. Fab.
74.	5292	Mooney	O: Si wafers	Opt. Fab.

QPR #6

75.	5293	Horrigan	O: Al laser mirror	Opt. Fab.
76.	5294	Hilton	O: InP 12:15:88	Opt. Fab.
77.	5295	Hilton	Q: Fused quartz disks	Material & Opt. Fab.
78.	5296	Hilton	O: InP (seed rods)	Opt. Fab.
79.	5297	Hilton	O: InP 12:15:88	Opt. Fab.
80.	5298	Harris	O: BSO #7 & #22	X-ray Serv.
81.	5299	Harris	O: BSO #17, #26 & #27 BGO #2	Opt. Fab.
82.	5300	Bachowski	O: InP 12:15:88 substrates	Material & Opt. Fab.

QPR #7

83.	5301	Hilton	O: InP 12:4	Opt. Fab.
84.	5302	Dumais	O: SrTiO ₃	X-ray Serv.
85.	5303	Harris	O: BSO	Opt. Fab.
86.	5304	Brown	G: 1360	Opt. Fab.
87.	5305	Dumais	O: Fused quartz	Material & Opt. Fab.
88.	5306	Ryan	O: Fused quartz	Opt. Fab.
89.	5307	Golubovic	O: Stainless steel	Opt. Fab.
90.	5308	Harris	O: BSO 32	X-ray Serv.
91.	5309	Hilton	O: InP #2-2	Opt. Fab.

QPR #8

92.	5310	Johnson	O: Unknown on InP	Auger
93.	5311	Weyburne	Q: Fused quartz	Opt. Fab.
94.	5312	Hilton	Q: Fused quartz (10)	Opt. Fab.

Material

QPR #9

95.	5313	Harris	O: BT4/BGO4/BSO/21-39	X-ray Serv. Opt. Fab.
96.	5314	Hilton	O: InP	X-ray Serv. Opt. Fab.
97.	5315	Harris	O: BT05/BSO/41	X-ray Serv. Opt. Fab.
QPR #10				
98.	5316	Harris	O: BSO (12 boules)	X-ray Serv. Opt. Fab.
99.	5317	Hilton	O: InP 11:14:89	X-ray Serv. seed plates
100.	5318	Harris	O: BGO-BSO (6 boules)	X-ray Serv. Opt. Fab.
101.	5319	Harris	O: BSO (3 boules)	X-ray Serv. Opt. Fab.
102.	5320	Mooney	O: Si-IRS (6) Si-CLD (1)	Opt. Fab.
103.	5321	Dumais	O: SEM Standard	Opt. Fab.
QPR #11				
104.	5322	Harris	O: BGO 11, BSO 56* & 57 (* 21.98 gr)	Opt. Fab.
105.	5323	Golubovic	Q: Fused quartz	Material Opt. Fab.
106.	5324	Spaziani	Q: Fused quartz	Opt. Fab.
107.	5325	Bliss	O: InP	Opt. Fab.
108.	5326	Harris	O: BTO 7/BSO 59/BS 27	Opt. Fab.
109.	5327	Harris	O: BSO 60 & 61	Opt. Fab.
110.	5328	Spaziani	Q: Fused quartz plates	Material Opt. Fab.
111.	5329	Spaziani	Q: Fused quartz prisms	Material Opt. Fab.
112.	5330	Spaziani	Q: Fused quartz prisms	Material Opt. Fab.
113.	5331	Hilton	Q: Fused quartz	Material Opt. Fab.
114.	5332	Bliss	Q: Fused quartz rod	Opt. Fab.
115.	5333	Spaziani	Q: Fused quartz	Opt. Fab.
116.	5334	Lipson	O: Si IRSD 19, 22A, 109, 111, 121	Opt. Fab.
117.	5335	Harris	O: BSO 64, BTO 8	X-ray Serv. Opt. Fab.
118.	5336	Spaziani	O: Fused quartz	Opt. Fab.

QPR #12--01 February 1990 to 30 April 1990,
incorporated as part of final report

Explanation/Table I

* The materials/specimen listed in this column are generalized:

Q= Quartz, as single crystal boules or fused quartz components

G= Experimental glass specimen, normally identified by lot or melt label.

O= Other, specimens of a mixed oxide crystal, a semi-conductor or a metal component.

** The services provided have been generalized:

Opt.Fab.= The preparation of a specimen to a critical surface finish/flatness/or dimension. In most cases, the letter report will provide detail as appropriate measurement records or data.

X-ray Serv.=X-ray diffraction measurement or record; Laue film, diffractometer 2θ recording or crystallographic measurement/calculation.

Mach.Serv.= Machining requests requiring the design or production of a fixture, a component, a fitting or an assembly.

END of FINAL REPORT